

## PATENT ABSTRACTS OF JAPAN

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### (54) PLASMA SPRAY COATING OF YTTRIA-STABILIZED ZIRCONIA

#### (57)Abstract:

PROBLEM TO BE SOLVED: To provide a YSZ plasma spray coating method to obtain YSZ(yttria- stabilized zirconia) sprayed coating high in adhesive strength and bending strength, furthermore low in a Young's modulus and excellent in thermal impact resistance.

SOLUTION: This plasma spray coating method to obtain yttria-stabilized zirconia sprayed coating is the one in which, on a base metal, raw material powder is plasma-sprayed, which is obtained by mixing 5 to 20 vol.% yttria- stabilized zirconia powder made into a hollow shape which is obtd. by gas atomization into yttria-stabilized zirconia powder which is obtd. by melting and pulverizing.

Moreover, on a base metal the latter yttria-stabilized zirconia powder is plasma-sprayed to have a thickness of 0.05 to 0.1 mm, and thereafter, the former yttria-stabilized zirconia powder is plasma-sprayed.

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## CLAIMS

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[Claim(s)]

[Claim 1] The plasma metal spray method of a yttria-stabilized-zirconia thermal-spraying coat for the adhesion reinforcement and flexural strength which are

characterized by carrying out the plasma metal spray of the raw material powder which comes to carry out 5-20 volume % mixing of the yttria-stabilized-zirconia gas spraying powder of a hollow configuration to yttria-stabilized-zirconia melting grinding powder at a base material having been large, and Young's modulus having been low moreover, and having excelled in thermal shock resistance.

[Claim 2] The plasma metal spray method of a yttria-stabilized-zirconia thermal-spraying coat the adhesion reinforcement and flexural strength which are characterized by carrying out the plasma metal spray of the yttria-stabilized-zirconia gas spraying powder of a hollow configuration after carrying out the plasma metal spray of the yttria-stabilized-zirconia melting grinding powder to a base material at 0.05-0.1mm thickness were large, and Young's modulus was moreover low excellent in thermal shock resistance.

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## DETAILED DESCRIPTION

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[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention carries out the plasma metal spray of the yttria-stabilized-zirconia (henceforth YSZ) powder to a base material front face,

adhesion reinforcement and flexural strength are large and Young's modulus is related with the plasma metal spray method make the YSZ thermal-spraying coat which was low excellent in thermal shock resistance form.

[0002]

[Description of the Prior Art] Although YSZ is used for heat insulation and heat-resistant coating from the former, the gas spraying powder made into the melting grinding powder and hollow configuration of YSZ as a raw material is used independently, respectively. Although adhesion reinforcement and flexural strength are high when a plasma metal spray is performed only using melting grinding powder, only the coat in which Young's modulus is also inferior to thermal shock resistance highly is obtained. Although Young's modulus is low excellent in thermal shock resistance when a plasma metal spray is performed to it only using gas spraying powder, only a coat with low adhesion reinforcement is obtained. The Young's modulus of the coat at the time of using by each conventional powder independent and performing a plasma metal spray, flexural strength, and adhesion reinforcement are shown in the after-mentioned table 1. in order to control the property of a coat conventionally -- melting grinding powder and gas spraying powder -- although plasma metal spray conditions, such as a plasma gas flow rate and a powder distributed gas flow rate, are adjusted in both cases -- these approaches -- for example, although Young's modulus falls 10% when melting grinding powder is used, it is difficult also for flexural strength or adhesion reinforcement to obtain the coat which has the optimal properties, such as to fall 10%.

[0003]

[Problem(s) to be Solved by the Invention] In view of the above-mentioned technical level, adhesion reinforcement and flexural strength of this invention are large, moreover its Young's modulus is low and it tends to offer the YSZ plasma metal spray method which can form the YSZ thermal-spraying coat which was excellent in thermal shock resistance.

[0004]

[Means for Solving the Problem] The adhesion reinforcement characterized by this invention carrying out the plasma metal spray of the raw material powder which comes to carry out 5-20 volume % mixing of the YSZ gas spraying powder of a hollow configuration to (1) YSZ melting grinding powder at a base material, The plasma metal spray method of a YSZ thermal-spraying coat flexural strength was large and Young's modulus was moreover low excellent in thermal shock resistance And after carrying out the plasma metal spray of the (2) YSZ melting grinding powder to a base material at 0.05 - 0.1mm thickness, (It is hereafter called the 1st invention) The adhesion reinforcement and flexural strength which are characterized by carrying out the plasma metal spray of the YSZ gas spraying powder of a hollow configuration are large, and, moreover, Young's modulus is the plasma metal spray method (henceforth the 2nd invention) of a YSZ thermal-spraying coat for having excelled in thermal shock resistance low.

[0005]

[Embodiment of the Invention] (The 1st invention) As :YSZ melting grinding powder, YSZ is fused and what generally ground the thing made to solidify in particle size of 10-45 micrometers with grinding equipment is used. What generally made melt which fused YSZ the particle size of 10-100 micrometers with the gas atomiser as YSZ gas spraying powder of a hollow configuration is used. In a plasma metal spray, when only YSZ melting grinding powder is used, a precise coat also with the high adhesion between adhesion particles is formed, but when only the YSZ gas spraying powder of a hollow configuration is used, many clearances are seen between adhesion particles and the coat with high thermal shock resistance with which Young's modulus fell is formed.

[0006] Then, it is what uses as the raw material powder of a plasma metal spray what mixed both powder in consideration of the above-mentioned property of each powder in the 1st invention. As shown also in Table 1 of the after-mentioned example 1, having made into five to 20 volume % the amount of the YSZ gas spraying powder of a hollow configuration mixed by YSZ melting grinding powder When the mixing percentage of the YSZ gas spraying powder of

a hollow configuration is five volume %, it compares only with the conventional YSZ melting grinding powder. Although Young's modulus can decrease about 10%, and flexural strength and adhesion reinforcement hardly fall, and Young's modulus can decrease about 20% when mixing percentage is 20 volume %, it is because it stops at whether flexural strength and adhesion reinforcement are slight and falling about 10%.

[0007] (The 2nd invention) Plasma metal spray conditions are the same as the above in the YSZ gas spraying powder list of YSZ melting grinding powder and a hollow configuration. Having set to 0.05-0.1mm coating thickness of the YSZ melting grinding powder by which a plasma metal spray is carried out first is based on the following reason. It is because Young's modulus will become high and thermal shock resistance will deteriorate, if the coating thickness of YSZ melting grinding powder is large, and is because the effectiveness using YSZ melting grinding powder will become small if the thickness is small. Moreover, generally the thickness formed with YSZ gas spraying powder among the hollow configurations by which a plasma metal spray is carried out to the degree is 0.5-1mm.

[0008]

[Example] The concrete example of this invention and the example of a comparison are given hereafter, and effectiveness of this invention is clarified further.

[0009] (Example 1) In this example 1, in order to give thermal shock resistance to the plasma metal spray coat excellent in reinforcement and adhesion, raw material powder used for YSZ melting grinding powder the powder ingredient of which 5-20 volume % mixing was done with the gas spraying powder of YSZ of a hollow configuration. Moreover, the gas atomized powder of YSZ of a hollow configuration carried out also about the thing outside the above-mentioned range, the thing for which only YSZ melting grinding powder was used, and the thing only using the YSZ gas spraying powder of a hollow configuration for the comparison.

[0010] The experimental result about the above-mentioned thermal spray material is shown in Table 1. The characteristic test was performed about Young's modulus, flexural strength, and adhesion reinforcement. Young's modulus and the thermal-spraying coat for flexural strength measurement were obtained as follows. That is, thickness:0.5mm thermal spraying of the aluminum was carried out on the flat surface of steel-for-general-structure (JIS SS400) plate:5Wx40Lx5Tmm, thermal spraying of the YSZ was carried out on it, it dipped in the NaOH solution after that, the YSZ coat was released off structural steel by melting aluminum layer, and each trial (Young's modulus, flexural strength) was presented with the coat. Young's modulus and flexural strength were measured for the taken-out coat by the three-point bending test method. Performing measurement at a room temperature, the crosshead passing speed of a testing machine is 0.5 mm/min. An adhesion strength test is JIS. Production of a sample and measurement were performed according to H8666. The structural steel which has a YSZ coat was pasted up using an epoxy resin as other structural steel and adhesives without stripping remaining as it is, i.e., YSZ, and the adhesion reinforcement was measured. Performing measurement at a room temperature, a speed of testing is 1 mm/min. Making thermal spraying at this time into a plasma metal spray, plasma conditions are plasma gas Ar:40l./min, and min+H<sub>2</sub>: 13l./min, and plasma-arc current :630A, plasma-arc electrical-potential-difference:79V, spray distance: It could be 120mm.

[0011] This result is shown in Table 1. As compared with (the example 3 of a comparison), Young's modulus can reduce only the conventional YSZ melting grinding powder about 10%, and Table 1 shows that most of flexural strength and adhesion reinforcement is not falling, when the mixing percentage of the YSZ gas spraying powder of a hollow configuration is five volume % (the example 1 of the 1st invention). Moreover, when the YSZ gas spraying powder of a hollow configuration is 20 volume % (the example 2 of the 1st invention), Young's modulus can decrease about 20% and it turns out whether flexural strength and adhesion reinforcement are slight and that it has stopped at having fallen about

10%. However, when the mixing percentage of gas spraying powder is under 5 volume % (example 1 of a comparison), there is almost no decline in Young's modulus, and when mixing percentage is more than 20 volume % (example 2 of a comparison), it does not serve as a coat which flexural strength and adhesion reinforcement fell about 20% or more, was excellent in reinforcement, and was excellent also in thermal shock resistance.

[0012]

[Table 1]

表 1

試験名	中空形状のYSZの ガス噴霧粉末混合率 (%)	曲げ強度 (kgf/mm <sup>2</sup> )	密着強度 (kgf/mm <sup>2</sup> )	ヤング率 (kgf/mm <sup>2</sup> )
比較例 1	3	6.5	7.9	3550
第1発明例 1	5	6.4	7.8	3200
第1発明例 2	20	6.1	7.2	2900
比較例 2	25	5.6	6.8	2800
比較例 3	0	6.6	8.0	3600
比較例 4	100	3.5	5.0	1600

[0013] (Example 2) In this example 2, the plasma metal spray was performed so that a plasma metal spray might be carried out to the thickness of 0.05mm using YSZ melting grinding powder and all coating thickness might turn into 1mm thickness after that in the beginning using the gas spraying powder of YSZ of a hollow configuration.

[0014] The experimental result about the thermal spray material obtained by the above-mentioned approach is shown in Table 2. The characteristic test was performed about adhesion reinforcement and thermal conductivity. An adhesion strength test is JIS. Production of a sample and measurement were performed



according to H8666. Adhesives are epoxy resins, measurement is performed at a room temperature and a speed of testing is 1 mm/min. The thermal-spraying coat for a thermal conductimetry trial was formed at the thickness of 1mm on the flat surface of steel-for-general-structure (JIS SS400) plate phi10x5Tmm. In order to measure the thermal conductivity of only a coat, before carrying out thermal spraying of the YSZ, 0.5mm thermal spraying of the aluminum in thickness was carried out, by dipping in a NaOH water solution, aluminum layer was melted and only the YSZ coat is taken out. About the taken-out coat, it is JIS. Thermal conductivity was measured according to R1611. Making thermal spraying at this time into a plasma metal spray, plasma conditions are plasma gas Ar:40l./, and min+H2.: It considered as 13l./ min, and plasma-arc current:630A, plasma-arc electrical-potential-difference:79V, and the spray distance of 120mm.

[0015] This result is shown in Table 2. From Table 2, the adhesion reinforcement of the coat by this invention is improving compared with the conventional coat which used only the YSZ gas spraying powder of a hollow configuration, and thermal conductivity shows the same value as the coat in a conventional method.

[0016]

[Table 2]

表 2

試験名	密着強度 (kgf/mm <sup>2</sup> )	熱伝導率 [W/(m・K)]	曲げ強度 (kgf/mm <sup>2</sup> )	ヤング率 (kgf/mm <sup>2</sup> )
従来例	5. 0	1. 2	3. 5	1 6 0 0
第2発明例	8. 0	1. 2	3. 7	1 8 0 0

[0017]

[Effect of the Invention] It becomes producible [ the coat which has high adhesion reinforcement and was excellent also in adiabatic with this invention ]. Thereby, the use life of the device member used by hot environments can be developed

remarkably. Therefore, it leads to reduction of the restoration cost of a coat, and a very big thing has the effectiveness on industry.

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## (54) 【発明の名称】 イットリア安定化ジルコニア被膜のプラズマ溶射法

## (57) 【要約】

【課題】 密着強度、曲げ強度が大きく、しかもヤング率が低く耐熱衝撃性に優れた Y S Z 溶射被膜を形成することができる Y S Z プラズマ溶射法に関する。

【解決手段】 ① イットリア安定化ジルコニア溶融粉砕粉末に中空形状のイットリア安定化ジルコニアガス噴霧粉末を 5 ～ 20 体積% 混合してなる原料粉末を母材にプラズマ溶射することを特徴とする密着強度、曲げ強度が大きく、しかもヤング率が低く、耐熱衝撃性に優れたイットリア安定化ジルコニア溶射被膜のプラズマ溶射法及び ② イットリア安定化ジルコニア溶融粉砕粉末を母材に 0.05 ～ 0.1 mm 厚さにプラズマ溶射した後、中空形状のイットリア安定化ジルコニアガス噴霧粉末をプラズマ溶射することを特徴とする密着強度、曲げ強度が大きく、しかもヤング率が低く耐熱衝撃性に優れたイットリア安定化ジルコニア溶射被膜のプラズマ溶射法。

## 【特許請求の範囲】

【請求項1】 イットリア安定化ジルコニア溶融粉砕粉末に中空形状のイットリア安定化ジルコニアガス噴霧粉末を5～20体積%混合してなる原料粉末を母材にプラズマ溶射することを特徴とする密着強度、曲げ強度が大きく、しかもヤング率が低く、耐熱衝撃性に優れたイットリア安定化ジルコニア溶射被膜のプラズマ溶射法。

【請求項2】 イットリア安定化ジルコニア溶融粉砕粉末を母材に0.05～0.1mm厚さにプラズマ溶射した後、中空形状のイットリア安定化ジルコニアガス噴霧粉末をプラズマ溶射することを特徴とする密着強度、曲げ強度が大きく、しかもヤング率が低く耐熱衝撃性に優れたイットリア安定化ジルコニア溶射被膜のプラズマ溶射法。

## 【発明の詳細な説明】

## 【0001】

【発明の属する技術分野】本発明は、イットリア安定化ジルコニア（以下、YSZという）粉末を母材表面にプラズマ溶射して、密着強度、曲げ強度が大きく、ヤング率が低く耐熱衝撃性に優れたYSZ溶射被膜を形成させるプラズマ溶射法に関する。

## 【0002】

【従来の技術】従来からYSZが断熱および耐熱コーティングに利用されているが、原材料としてYSZの溶融粉砕粉末や中空形状にしたガス噴霧粉末が、それぞれ単独で用いられている。溶融粉砕粉末のみを用いてプラズマ溶射を行った場合、密着強度、曲げ強度が高いものの、ヤング率も高く耐熱衝撃性に劣る被膜しか得られない。それに対し、ガス噴霧粉末のみを用いてプラズマ溶射を行った場合、ヤング率が低く耐熱衝撃性に優れるが、密着強度の低い被膜しか得られない。後記表1に、従来の各粉末単独で用いてプラズマ溶射を行った場合の被膜のヤング率、曲げ強度、密着強度を示す。従来、被膜の特性を制御するために、溶融粉砕粉末、ガス噴霧粉末どちらの場合もプラズマガス流量や粉末供給ガス流量等のプラズマ溶射条件を調整しているが、これらの方法では例えば溶融粉砕粉末を用いた場合、ヤング率が10%低下するが、曲げ強度や密着強度も10%低下してしまうなど、最適な特性を有する被膜を得るのが難しい。

## 【0003】

【発明が解決しようとする課題】本発明は上記技術水準に鑑み、密着強度、曲げ強度が大きく、しかもヤング率が低く、耐熱衝撃性の優れたYSZ溶射被膜を形成することができるYSZプラズマ溶射法を提供しようとするものである。

## 【0004】

【課題を解決するための手段】本発明は（1）YSZ溶融粉砕粉末に中空形状のYSZガス噴霧粉末を5～20体積%混合してなる原料粉末を母材にプラズマ溶射することを特徴とする密着強度、曲げ強度が大きく、しかも

ヤング率が低く耐熱衝撃性に優れたYSZ溶射被膜のプラズマ溶射法（以下、第1発明という）及び（2）YSZ溶融粉砕粉末を母材に0.05～0.1mm厚さにプラズマ溶射した後、中空形状のYSZガス噴霧粉末をプラズマ溶射することを特徴とする密着強度、曲げ強度が大きく、しかもヤング率が低く耐熱衝撃性に優れたYSZ溶射被膜のプラズマ溶射法（以下、第2発明という）である。

## 【0005】

【発明の実施の形態】（第1発明）：YSZ溶融粉砕粉末としては、YSZを溶融し、凝固させたものを粉砕装置で一般的には10～45μmの粒径に粉砕したものが使用される。中空形状のYSZガス噴霧粉末としては、YSZを溶融した溶融物をガス噴霧装置で一般的には10～100μmの粒径にしたものが使用される。プラズマ溶射において、YSZ溶融粉砕粉末のみを用いた場合は、付着粒子間の密着性も高く緻密な被膜が形成されるが、中空形状のYSZガス噴霧粉末のみを用いた場合は、付着粒子間に隙間が多くみられ、ヤング率の低下した耐熱衝撃性の高い被膜が形成される。

【0006】そこで、第1発明ではそれぞれの粉末の上記特性を考慮して、両粉末を混合したものをプラズマ溶射の原料粉末とするものであって、YSZ溶融粉砕粉末に混合される中空形状のYSZガス噴霧粉末の量を5～20体積%としたのは後記実施例1の表1にも示したように、中空形状のYSZガス噴霧粉末の混合率が5体積%の場合、従来のYSZ溶融粉砕粉末のみと比較して、ヤング率が約10%低減でき、かつ曲げ強度、密着強度は殆ど低下せず、また混合率が20体積%の場合、ヤング率が約20%も低減できるにも拘らず、曲げ強度、密着強度は僅か約10%低下するに留まるからである。

【0007】（第2発明）YSZ溶融粉砕粉末、中空形状のYSZガス噴霧粉末並びにプラズマ溶射条件は上記と同じである。先ずプラズマ溶射されるYSZ溶融粉砕粉末の被膜厚さを0.05～0.1mmとしたのは、下記理由による。YSZ溶融粉砕粉末の被膜厚さが大きいと、ヤング率が高くなり、耐熱衝撃性が劣化するからであり、またその厚さが小さいとYSZ溶融粉砕粉末を用いる効果が小さくなるからである。また、その次にプラズマ溶射される中空形状のうちYSZガス噴霧粉末によって形成される膜厚は一般的に0.5～1mmである。

## 【0008】

【実施例】以下、本発明の具体的な実施例、比較例をあげ、本発明の効果を一層明らかにする。

【0009】（例1）この例1においては、強度、密着性に優れたプラズマ溶射被膜に、耐熱衝撃性を付与するために、原料粉末はYSZ溶融粉砕粉末に中空形状のYSZのガス噴霧粉末で5～20体積%混合した粉末材料を用いた。また、比較のために、中空形状のYSZのガ

ス噴霧粉が上記範囲外のもの、YSZ溶融粉砕粉末のみを用いたもの及び中空形状のYSZガス噴霧粉末のみを用いたものについても行った。

【0010】表1に、上記溶射材料についての実験結果を示す。特性試験はヤング率、曲げ強度及び密着強度に関して行った。ヤング率および曲げ強度測定用の溶射被膜は次のようにして得た。すなわち、一般構造用鋼（JIS SS400）板：5W×40L×5Tmmの平面上にA1を厚さ：0.5mm溶射し、その上にYSZを溶射し、その後NaOH溶液に浸し、A1層を溶かすことでYSZ被膜を構造用鋼からはなし、その被膜を各試験（ヤング率、曲げ強度）に供した。取り出した被膜を3点曲げ試験法によりヤング率および曲げ強度を測定した。測定は室温で行い、試験機のクロスヘッド移動速度は0.5mm/minである。密着強度試験はJIS H8666に準じて、試料の作製、測定を行った。YSZ被膜を有する構造用鋼をそのまま、すなわちYSZをはがすことなく、他の構造用鋼と接着剤としてエポキシ樹脂を用いて接着し、その密着強度を測定した。測定は室温で行い、引張速度は1mm/minである。このと

表 1

試験名	中空形状のYSZの ガス噴霧粉末混合率 (%)	曲げ強度 (kgf/mm <sup>2</sup> )	密着強度 (kgf/mm <sup>2</sup> )	ヤング率 (kgf/mm <sup>2</sup> )
比較例1	3	6.5	7.9	3650
第1発明例1	5	6.4	7.8	3200
第1発明例2	20	6.1	7.2	2900
比較例2	25	5.6	6.8	2800
比較例3	0	6.6	8.0	3600
比較例4	100	3.5	5.0	1600

【0013】（例2）この例2においては、初めはYSZ溶融粉砕粉末を用いて0.05mmの厚さにプラズマ溶射し、その後、中空形状のYSZのガス噴霧粉末を用いて全被膜厚さが1mm厚さになるように、プラズマ溶射を行った。

【0014】表2に、上記方法によって得られた溶射材料についての実験結果を示す。特性試験は密着強度および熱伝導率に関して行った。密着強度試験はJIS H8666に準じて、試料の作製、測定を行った。接着剤はエポキシ樹脂であって測定は室温で行い、引張速度は1mm/minである。熱伝導率測定試験用の溶射被膜は、一般構造用鋼（JIS SS400）板φ10×5

きの、溶射はプラズマ溶射とし、プラズマ条件はプラズマガスAr：40リットル/min+H<sub>2</sub>：13リットル/min、プラズマアーク電流：630A、プラズマアーク電圧：79V、溶射距離：120mmとした。

【0011】この結果を表1に示す。表1より、中空形状のYSZガス噴霧粉末の混合率が5体積%の場合（第1発明例1）、従来のYSZ溶融粉砕粉末のみ（比較例3）と比較してみると、ヤング率が約10%低減でき、かつ、曲げ強度、密着強度はほとんど低下していないことが分かる。また、中空形状のYSZガス噴霧粉末が20体積%の場合（第1発明例2）、ヤング率が約20%低減でき曲げ強度、密着強度は僅か約10%低下したに留まっていることが分かる。しかし、ガス噴霧粉末の混合率が5体積%未満の場合（比較例1）、ヤング率の低下は殆どなく、又混合率が20体積%以上の場合（比較例2）、曲げ強度、密着強度が約20%以上低下してしまい、強度に優れた耐熱衝撃性にも優れた被膜とはならない。

【0012】

【表1】

Tmmの平面上に、1mmの厚さに形成した。被膜のみの熱伝導率を測定するため、YSZを溶射する前に、A1を厚さ0.5mm溶射し、NaOH水溶液に浸すことで、A1層を溶かし、YSZ被膜のみを取り出している。取り出した被膜を、JIS R1611に準じて、熱伝導率の測定を行った。このときの、溶射はプラズマ溶射とし、プラズマ条件はプラズマガスAr：40リットル/min+H<sub>2</sub>：13リットル/min、プラズマアーク電流：630A、プラズマアーク電圧：79V、溶射距離120mmとした。

【0015】この結果を表2に示す。表2より、本発明による被膜の密着強度は中空形状のYSZガス噴霧粉末

のみを用いた従来の被膜と比べて向上しており、かつ、【0016】  
熱伝導率は従来法での被膜と同じ値を示している。【表2】

表 2

試験名	密着強度 (kgf/mm <sup>2</sup> )	熱伝導率 [W/(m・K)]	曲げ強度 (kgf/mm <sup>2</sup> )	ヤング率 (kgf/mm <sup>2</sup> )
従来例	5.0	1.2	3.5	1600
第2発明例	8.0	1.2	3.7	1800

【0017】  
【発明の効果】本発明によって、高い密着強度を有しか  
つ断熱性にも優れた被膜の作製が可能となる。これによ

り、高温環境で使用する機器部材の使用寿命を著しく伸  
ばすことができる。従って、被膜の修復コストの低減に  
繋がり、産業上の効果は非常に大きなものがある。